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IS 4023 (1991): Methods for the determination of reactivity of coke [PCD 7: Solid Mineral Fuels]

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कोक अभिक्रियाशीलता ज्ञात करने की पद्धतियों का मसौदा

(पहला पुनरीक्षण)

Indian Standard

METHODS FOR THE DETERMINATION OF REACTIVITY OF COKE

(*First Revision*)

UDC 662·749·3 : 543·878

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1991

Price Group 4

Solid Mineral Fuels Sactional Committee, PCD 7

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Solid Mineral Fuels Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Reactivity of coke to carbon dioxide (carboxy-reactivity), steam (hydro-reactivity), oxygen (oxy-reactivity) and sulphur (thio-reactivity) has been studied in detail and the importance of reactivity to users of coke is now widely appreciated. It is probable that the various reactivities determined in the laboratory on small coke particles are not strictly correlated to the behaviour of lump coke in gas producers, cupolas and blast furnaces, as various other factors also exert their influence. However, a comparison of the reactivity as determined in the laboratory of a number of samples of different reactivities of coke, shows whether a particular coke is reactive in comparison to the others under similar conditions. The factor showing the greatest correlation with reactivity under identical conditions of precarbonization and carbonization appears to be the average rank of the parent coals as measured by their average reflectance or volatile matter content on a dry basis.

This standard prescribes methods of test for reactivity of coke in a comprehensive manner wherein (a) reactivity of coke to air [also called critical air blast (CAB)], and (b) reactivity of coke to carbon dioxide (RCO_2) are dealt with. The Sectional Committee responsible for preparation of this standard felt that the method for determination of thermal value of volatiles is not very essential and hence the same has been excluded from this version.

Critical air blast (CAB) is the minimum rate of air blast which will maintain combustion of closely graded fuel in an ignited bed of specified dimensions. The more reactive the coke, the lower is its CAB value.

Reactivity of coke to carbon dioxide (RCO_2) is denoted by the volume in millilitres of carbon monoxide (at normal temperature and pressure) formed from 100 ml of carbon dioxide (also at normal temperature and pressure) on passing through a specified column of heated coke under standard conditions. The more reactive the coke, the higher is its RCO_2 value. Aternatively, the percentage loss after reaction of coke with carbon dioxide is reckoned as coke reactivity index (CRI). The coke strength after reaction is also measured to give the CSR value.

In the formulation of this standard, due weightage has been given to international co-ordination among the standards and practices prevailing in different countries in addition to relating it to the practices in the field in this country.

In reporting the result of a test made in accordance with this standard, if the final value, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR THE DETERMINATION OF REACTIVITY OF COKE

(First Revision)

1 SCOPE

1.1 This standard prescribes the methods of test for reactivity of coke, namely, (a) reactivity of coke to air [critical air blast (CAB)], (b) reactivity of coke to carbon dioxide (RCO_2) or coke reactivity index (CRI), and (c) coke strength after reaction (CSR):

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
266 : 1977	Sulphuric acid (*second revision*)
436 (Part 2) : 1965	Methods of sampling of coal and coke : Part 2 Sampling of coke (*revised*)
1070 : 1977	Water for general laboratory use (*second revision*)
1314 : 1984	Calcium chloride (*second revision*)

3 DETERMINATION OF CRITICAL AIR BLAST

3.1 Outline of the Method

In a standard electrically heated combustion chamber pass dry air at the rate of 4·2 l/min (or 0·004 2 m^3/min) over a bed of graded coke till a glow appears. When the coke is fully ignited the rate of passing air is reduced to a preselected value for minutes and then raised to the original value to attempt resuscitation. This procedure is repeated with fresh portions of the coke with other rates of air flow until the critical value is found.

3.2 Apparatus

3.2.1 *Combustion Chamber*

It consists of a transparent, fused silica or heat-resisting glass tube 20 cm long, having internal diameter of 40 ± 1 mm and wall thickness of 1 to 2 mm. It is provided with three fused-on lugs, or indentations, on the inside of the wall of the tube 35 to 41 mm from one end. A continuous line shall be marked round the circumference 10 to 12 mm from the same end. It is closed at the other end by a rubber stopper which carries a silica or heat-resisting glass tube of 5 to 6 mm internal diameter bent at right angles.

3.2.2 *Metal Gauge Disk*

Made from wire cloth having aperture of 425 to 600 microns to fit inside the combustion chamber and rest on the lugs or indentations.

3.2.3 *Heating Element*

A cylindrical frame of insulating and heat resisting material of 50 mm outside diameter, 39 mm internal diameter and 20 mm long, recessed so as to rest on the top of the combustion chamber (Fig. 1). The diameter of the recess shall be 1 to 4 mm greater than that of the combustion chamber. The heating wire, which shall be of 0·3 mm diameter thickness nickel-chromium resistance wire welded at its ends to 0·9 mm thickness leads of the same alloy. It shall be wound helically round and supported by five silica rods, 2·5 ± 0·25 mm outside diameter, fitting into holes drilled through the walls of the frame. The resistance of the wire (when new), as measured between the leads not exceeding 50 mm in length, shall be between 38 to 40 ohms. The top of the frame shall be recessed to approximately 46 mm diameter and 3 mm deep to take a porcelain or quartz disk with at least 50 holes of diameter between 2 and 0·5 mm.

3.2.4 *Air Control System*

Air may be supplied from a cylinder of compressed air or directly from a compressor. In the latter case, a large reservoir should be included to damp out pulsations and a filter to remove oil mist, if necessary. The rate of air flow through the apparatus is controlled by a needle valve or screw clip placed after some form of pressure regulator, such as a water seal (up to 30 cm deep) or a gas governor.

3.2.5 *Measurement of Air Flow*

The following are suitable.

3.2.5.1 Two flow meters, one inclined capable of measuring flow rates up to 2·8 l/min (or

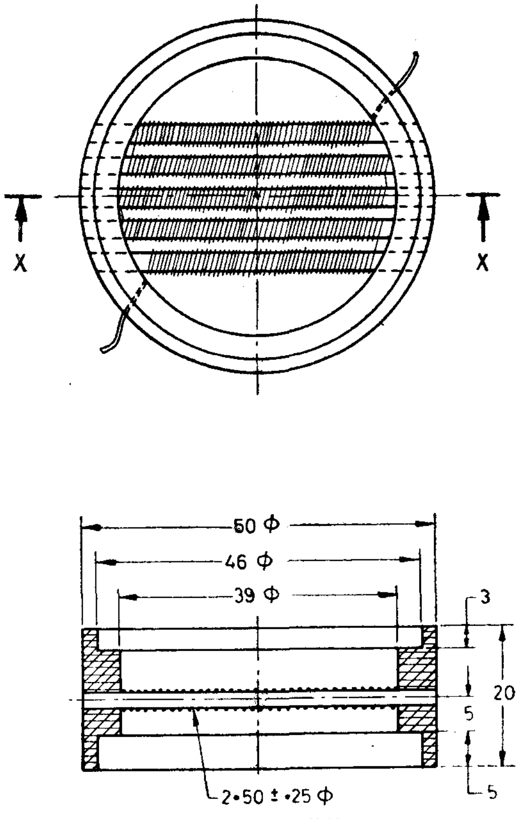
0·002 8 m^3/min) with an accuracy of 0·30 l/min (or 0·000 30 m^3/min) and the other to read 4·5 l/min (or 0·004 5 m^3/min.

3.2.5.2 An inclined capillary flow gauge, a gas meter 2·0 l/min (or 0·002 m^3/min) and a suitable manometer.

A typical flow diagram for measurement of air flow, etc, is shown in Fig. 2.

3.2.5.3 *Dome*

A dome of heat-resisting glass, 75 ± 15 mm internal diameter and 190 ± 10 mm high (Fig. 3A).



All dimensions in millimetres.

FIG. 1 DETERMINATION OF CRITICAL AIR BLAST — HEATING ELEMENT

3.2.5.4 A tube, of similar material and dimensions, closed at its upper end with a rubber stopper carrying the air inlet tube (Fig. 3B). The lower end of either is closed with a rubber stopper which carries the combustion chamber, brass or copper leads, 3·17 mm diameter, for making connection with the leads of heating element and the air inlet tube in the case of **3.2.5.3**.

3.2.5.5 *Drying tower*

25 cm high and 50 mm in diameter with tubular bottom filled with self-indicating silica gel.

3.2.5.6 *Funnel*

For charging the fuel, made by rolling a sheet of paper or metal foil into a cone having an outlet at the apex of 9·5 to 12·5 mm diameter.

3.2.5.7 *Former*

Of cork, felt covered wood or aluminium, of a diameter slightly less than that of the combustion chamber.

3.3 Sample Preparation

The coke used for the determination of critical air blast is the sample of coke, graded to pass a 1·18 mm IS sieve and to be retained on 600 micron IS sieve, taken and prepared according to IS 436 (Part 2) : 1965. The coke shall be dried in a shallow tray in an air oven at 105°C to 110°C and resieved on a 600-micron IS sieve immediately before use.

3.4 Procedure

3.4.1 Assemble the apparatus as shown in Fig. 2 and 3. Prepare the coke bed by pouring the resieved dry coke on the bed support, through the funnel, which shall be removed around so as to form a loose and uniformly packed bed. The height of the funnel above the fuel bed shall be such that the free fall of the particles does not exceed 5 mm. Fill the combustion chamber with fuel up to the line. Smooth the surface of the bed with the former, so manipulated as to roll the particles over the surface than to press them in position. Reassemble the apparatus and check for leaks.

3.4.2 Set the air blast at 4·2 l/min (or 0·004 2 m^3/min), switch on the current and adjust it so that the heat dissipated is between 175 and 185 watts. Continue heating until the glowing zone has spread substantially to all parts of the periphery of the bed surface. At this instane, switch off the current and reduce the blast rate to a value which is thought to be slightly above the critical (Note 1). Continue passing air at this rate for 20 minutes, and increase the rate to 4·5 l/min (or 0·004 5 m^3/min. Continue at this rate for 20 minutes or till resuscitation occurs. If resuscitation takes place, repeat the test using progressively lower air blast rate, with a fresh portion of sample for each new rate, until a rate is found at which no resuscitation occurs. Similarly, determine the air blast rate at which resuscitation just occurs (Note 2).

NOTES

1 A rough estimate of the CAB may be obtained from the time required for ignition.

2 Observations are made easier by screening the combustion chamber from direct lighting,

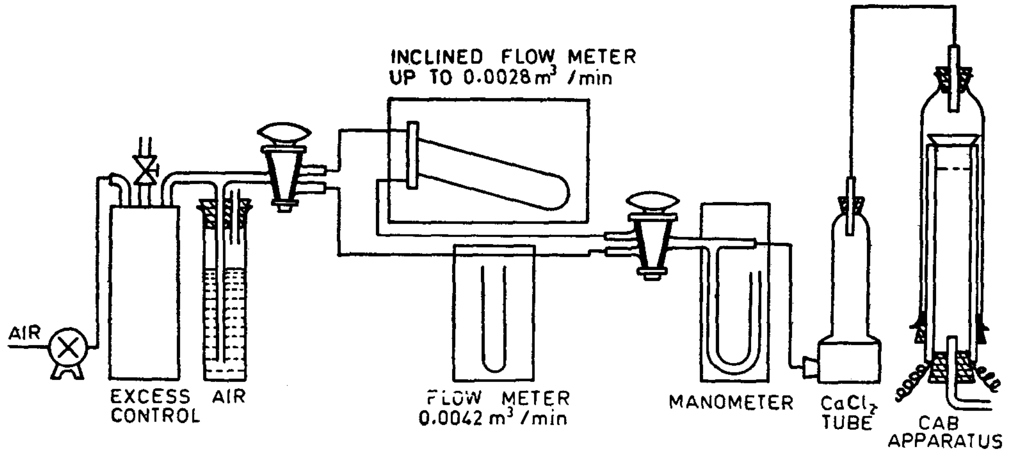
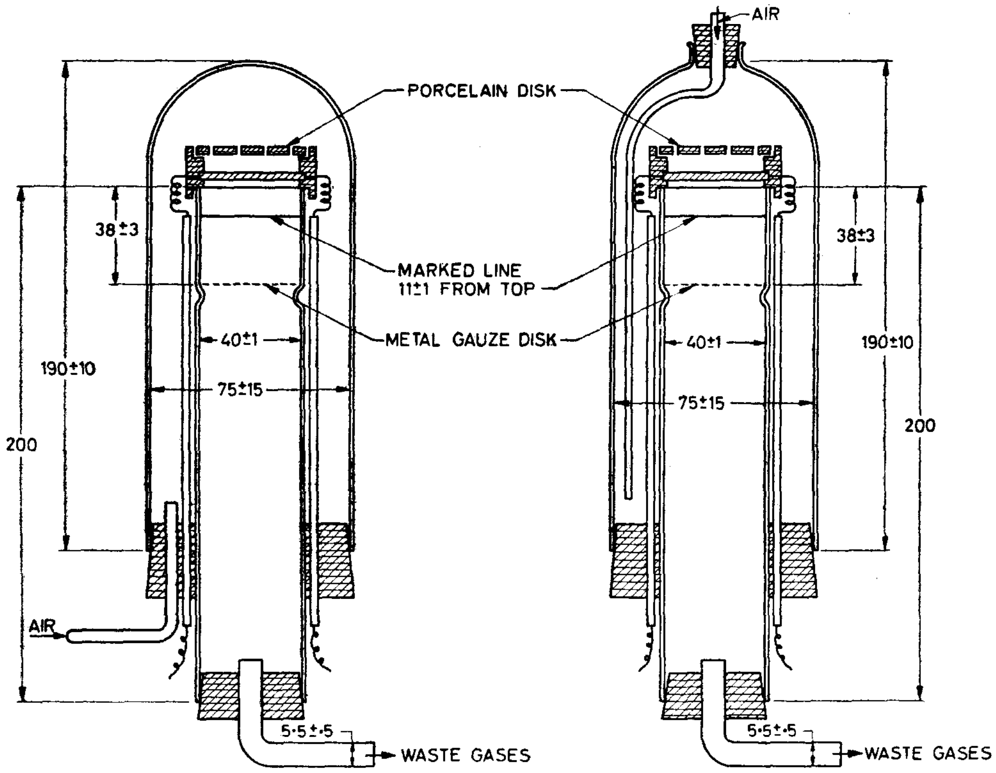


FIG. 2 TYPICAL FLOW DIAGRAM FOR MEASURING AIR FLOW IN DETERMINATION OF CRITICAL AIR BLAST



3A Bottom Inlet

3B Top Inlet

All dimensions in millimetres.

FIG. 3 DETERMINATION OF CRITICAL AIR BLAST — ALTERNATIVE ASSEMBLIES OF DOME AND COMBUSTION CHAMBER

In this way, obtain two rates of air blast which differ by not more than 0·05 l/min (or 0·000 05 m^3/min) and are such that resuscitation occurs with one but not with the other. The CAB is that value at which resuscitation occurs.

3.5 Calculation

$$\text{Critical Air Blast (CAB)} = \frac{532{\cdot}2\, PV}{(273 + T)\, d} \text{ when air is measured by flow meter}$$

where

P = atmospheric pressure (mm Hg);

V = air blast rate, at which resuscitation occurs (kl/min of dry air);

T = air temperature (°C); and

d = diameter of combustion chamber (mm).

4 DETERMINATION OF REACTIVITY OF COKE TO CARBON DIOXIDE

4.0 Two methods for determination of reactivity of coke to carbon dioxide have been prescribed, namely, Method A and Method B.

4.1 Method A

4.1.0 *Outline of the Method*

A standard reaction tube, containing graded coke sample, is heated from room temperature to the specified reaction temperature. After attaining standard conditions, 100 ml of carbon dioxide is passed at a definite rate through the coke bed and the volume of carbon monoxide formed is measured to give the reactivity value.

4.1.1 *Standard Conditions*

The standard conditions to be maintained for the determination of reactivity of coke shall be as follows:

a) Temperature of coke	950 ± 2°C
b) Rate of passage of carbon-dioxide	5 ml per minute
c) Volume of carbon dioxide to be passed for each determination	100 ml
d) Length of column of coke in reaction tube (with thermocouple in position)	7·5 cm
e) Size of coke	Between 0·6 and 1·2 mm
f) Normal temperature	0°C (273°K)
g) Normal pressure	760 mm Hg (760 × 133·322 N/m^2)

4.1.2 *Apparatus*

The working arrangement of the apparatus is illustrated in Fig 4. The set-up consists of the following main constituents.

4.1.2.1 *Preheater furnace*

Maintained at 600 ± 2 °C and containing copper for removing any oxygen in the gases and preheating prior to entering the reaction tube.

4.1.2.2 *Gas washers*

Two, one containing alkaline pyrogallol solution and the other concentrated sulphuric acid.

4.1.2.3 *Gas driers*

Three, containing concentrated sulphuric acid, calcium chloride and phosphoric oxide respectively.

4.1.2.4 *Reservoir*

Containing concentrated sulphuric acid and arranged so that the whole apparatus can be left under pressure of nitrogen whilst both the furnace and reaction tube are being cooled. It also enables the pyrogallol wash solution to be changed without disturbing other parts of the apparatus.

4.1.2.5 *Marriot bottle*

Containing concentrated magnesium chloride solution for expelling carbon dioxide at a constant rate of flow into the side tube. This bottle is fitted with a capillary air tube and a jet so arranged that it delivers approximately 5 ml of gas per minute. The jet is connected to the bottle by means of a bent glass tube and a short length of rubber tubing. By movement of the jet in the tubing the head of liquid can be varied and the rate of flow altered (*see* Note).

NOTE — This method of variation has been found to be much easier to control than a tap placed in the system.

4.1.2.6 *Reaction tube furnace*

Having a tube, 30 cm in length and 5 cm in in internal diameter, and wound on the outside with nichrome wire so that a uniform temperature of 950 ± 2°C might be attained at least through a length of 8 cm in the middle portion. The reaction tube is made of silica. A piston of steatite just slides into the silica tube and a longer silica capillary tube, also a sliding fit, is secured to the silica sheath by a packing of asbestos or kieselguhr. The whole assembly is fitted into the reaction tube by means of the rubber bung containing two holes one (central) for the sheath, and the other for the small phosphoric oxide tube. On the outside of the reaction tube three lines are drawn (with ferric chloride solution) at distances of 3·75 cm, 7 cm and 7·5 cm from the square shoulder where the capillary joins the main tube, (*see* Note). The other end of the reaction tube is a silica capillary 7 to 8 mm external diameter and 1·5 mm bore, joined to the larger tube with a square shoulder.

NOTE — The mark at 3·75 cm indicates the position of the tip of the thermocouple.

4.1.2.7 *Thermocouples*

Made of platinum-rhodium and platinum, and provided with suitable millivoltmeter for computing temperature. The thermocouple group is built round the silica thermocouple sheath about 5 mm in external diameter.

4.1.2.8 *Nitrometer*

Connected to the silica capillary through a three-way tap for collecting and measuring the volume of carbon monoxide formed.

4.1.2.9 *Kipp's apparatus*

For producing carbon dioxide.

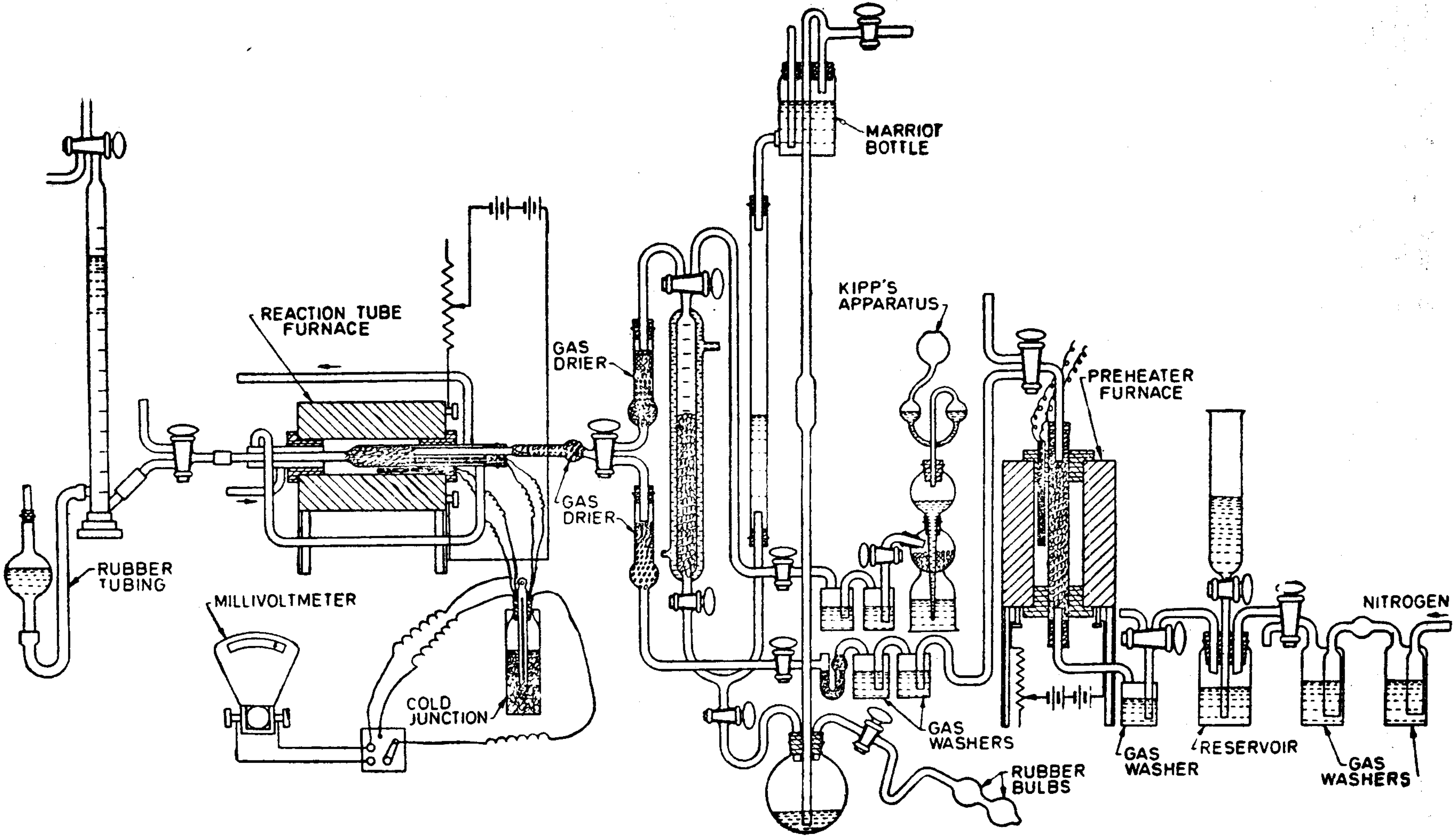


FIG. 4 APPARATUS FOR DETERMINING REACTIVITY OF COKE

4.1.3 *Reagents*

4.1.3.1 *Quality of reagents*

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be used in the test (*see* Note).

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4.1.3.2 *Reagents required*

a) *Nitrogen gas* — cylinder nitrogen, purified by washing with alkaline pyrogallol solution and dried with concentrated sulphuric acid before entering and after passing through the preheater furnace.

b) *Alkaline pyrogallol solution* — prepared by mixing 30 percent pyrogallic acid (*m*/*v*) solution and 60 percent (*m*/*v*) solution of potassium hydroxide in the proportion 1 : 3·5.

e) *Concentrated sulphuric acid* — *see* IS 266 : 1977.

d) *Calcium chloride* (*anhydrous*) — *see* IS 1314 : 1967.

e) *Phosphoric oxide*

f) *Carbon dioxide* — prepared in Kipp's apparatus using hydrochloric acid and selected marble chippings (previously boiled with water for several hours). The gas is washed with sodium carbonate solution and dried by passing through concentrated sulphuric acid.

g) *Sodium carbonate solution* — 10 percent (*m*/*v*) solution.

h) *Concentrated magnesium chloride solution* — saturated at room temperature.

j) *Potassium hydroxide solution* — 40 percent (*m*/*v*) for use in the nitrometer.

4.1.4 *Procedure*

Insert a small pad of asbestos into the reaction tube and fill it with the prepared sample of coke up to the 7 cm mark, tapping the end of the tube lightly to settle the grains of the sample until no more can be added. Insert the thermocouple sheath into the reaction tube through the rubber bung. The level of sample in the tube rises to 7·5 cm mark due to the displacement of coke by the sheath and shows the apparent volume of coke when the thermocouple is in position. Then set up the apparatus.

Raise the temperature of the reaction tube (containing coke sample) from room temperature to 950±2°C in a current of nitrogen duly purified by washing with alkaline pyrogallol solution and drying with concentrated sulphuric acid, and preheated to 600±2°C, about 1 000 ml of nitrogen and 1 hour being taken for this. Maintain the temperature of the reaction tube at 950±2°C for another 1 hour and continue to pass nitrogen at the same rate. Stop nitrogen flow and then pass 100 ml of carbon dioxide [*see* **4.1.3.2** (f)] prepared in the Kipp's apparatus at the standard rate of flow [*see* **4.1.1**(b)] before starting the determination. Start passing another 100 ml of carbon dioxide and collect the issuing gas in the nitrometer (**4.1.2.8**) over potassium hydroxide solution.

4.1.5 *Calculation*

$$\text{Reactivity of coke} = \frac{V_1}{V_2} \times 100$$

where

V_1 = volume in millilitres at NTP of carbon monoxide in the nitrometer as estimated by means of gas analyser.

V_2 = volume in millilitres at NTP of carbon dioxide passed through the reaction tube.

4.2 Method B

4.2.0 *Outline of the Method*

The sampled and sized coke pieces are charged into a reaction tube and heated to 1 100°C in a stream of nitrogen/argon. Carbon dioxide is then passed at the rate of 5 l/min for 120 minutes. The loss of mass after reaction is a measure of the rate of reaction of coke with carbon dioxide represented by $C + CO_2 = 2\,CO$.

After reaction the coke loses its strength and to assess the loss in strengh the reacted pieces are charged into a I-type drum which is rotated at a specified speed. The coke pieces are screened and the + 10 mm fraction taken as a measure of the strength after reaction.

4.2.1 *Standard Conditions*

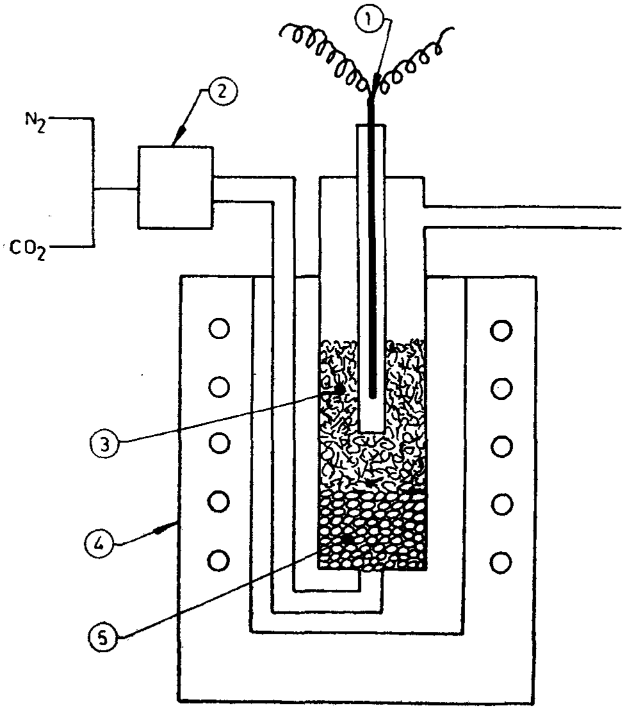
The test conditions shall be as follows:

a) Size of coke	19 to 21 mm
b) Sample mass	200 g
c) Test temperature	1 100°C ± 5°C
d) Max rate of flow of nitrogen/argon during heating and cooling	5 l/min
e) Heating time	45 min (*Max*)
f) Soaking time at 1 100°C	15 min
g) Rate of flow of carbon dioxide	5 l/min

h) Reaction time with carbon dioxide	120 min
j) Revolution of I-drum	30 min at 20 rpm
k) Screening after rotation	10 mm (round hole) screen
m) Dimension of I-type drum after tester	130 mm dia × 700 mm length

4.2.2 *Reactivity Apparatus*

The apparatus is illustrated in Fig 5. The details of the essential components are as follows:



1 Thermocouple
2 Gas Flow Meter
3 Sample
4 Electric Furnace
5 Alumina Balls

FIG. 5 APPARATUS FOR MEASURING COKE REACTIVITY INDEX

4.2.2.1 *Furnace*

Electrical resistance furnace maintainable at around 1 150°C, so as to obtain 1 100 ± 5°C in the reaction tube shall be used.

4.2.2.2 *Reaction tube*

The tube shall preferably be made of good heat resistance material like Inconncl-600. The internal diameter should be 76 mm. A 50 mm deep layer of refractory (such as fire clay) balls, 10 mm in diameter, shall be provided for proper distribution of gases. A thermocouple shall be inserted to the centre of the coke bed. The inlet and outlet pipes shall also be made of similar heat resistant material.

4.2.2.3 *Gas quality*

The inert gases (nitrogen/argon) shall be free of oxygen, moisture, carbon dioxide. A calcium chloride moisture trap shall be used. The reaction gas, that is, carbon dioxide shall be free from oxygen and moisture. The purity shall be 99 percent *Min.* A similar moisture trap shall be used.

4.2.2.4 *Flowmeter*

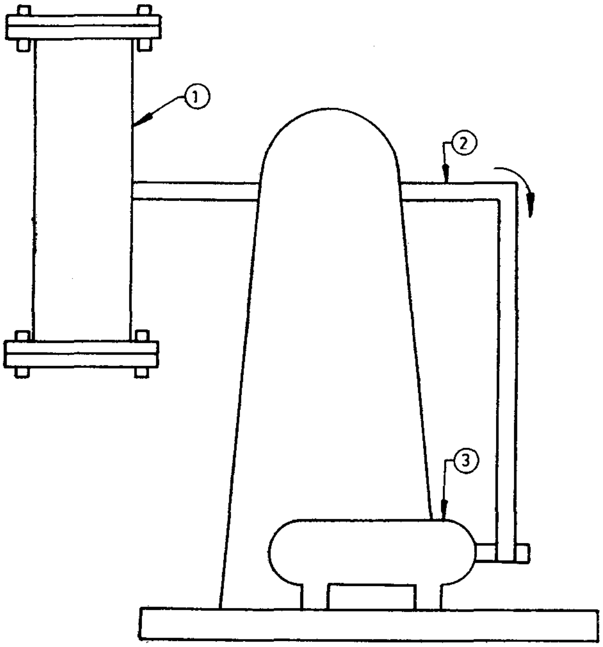
A standard rotameter or any other standard flow measuring device with proper control valve shall be included in the circuit.

4.2.2.5 *Safety measurement*

The exhaust gas from the reaction tube contains carbon monoxide and it should be either burnt by a pilot flame or disposed off at a suitable location away from the laboratory.

4.2.2.6 *I-Type drum tester*

A sketch is shown in Fig. 6. Necessary dimensions are given in **4.2.1.**



1 Drum 2 Belt 3 Motor

FIG. 6 I-TYPE DRUM TESTER FOR MEASURING COKE STRENGTH

4.2.3 *Procedure*

The sampled coke pieces are dried thoroughly to constant weight at 105°C and charged into the reaction tube. The thermocouple is inserted at the proper location i.e. middle of the coke bed. The reaction tube is placed in the furnace with nitrogen/argon switched on to prevent oxidation of coke pieces during heating. A soaking time of 15 min is allowed after the specified reaction temperature of 1 100°C is reached. Nitrogen flow is stopped and carbon dioxide is then passed at 5 l/min for 120 minutes maintaining the temperature at 1 100 ± 5°C. After this carbon dioxide is stopped, furnace is switched off and cooling to room temperature is done in a stream of nitrogen/argon. Coke pieces are carefully withdrawn and weighed. The coke is transferred to the drum and rotated for 30 min at 20 rpm. The sample is then taken out and screened on a 10 mm round hole sieve.

4.2.4 *Calculation*

Coke Reactivity Index (CRI)

$$= \frac{200 - W_1}{200} \times 100$$

Coke Strength after Reaction (CSR)

$$= \frac{W_2}{W_1} \times 100$$

where

W_1 = mass of coke in g after reaction; and

W_2 = mass of + 10 mm in g coke after rotation in I-type drum.

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BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices : Telephone

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002 — 331 01 31, 331 13 75

Eastern : 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola
CALCUTTA 700054 — 87 86 62

Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036 — 53 38 43

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